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Quality Assurance and Quality Control Data Validation Procedures Used for the Love Canal and Dallas Lead Soil Monitoring Programs

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QUALITY ASSURANCE AND QUALITY CONTROL DATA VALIDATION PROCEDURES USED FOR THE LOVE CANAL AND DALLAS LEAD SOIL MONITORING PROGRAMS

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Abstract. Public awareness of soils contamination has increased in recent years due in part to the notoriety associated with the indiscriminate release, packaging, transporting and disposal of hazardous materials. In 1980, and again in 1982, the U.S. Environmental Protection Agency (EPA) was responsible for designing, implementing and conducting environmental monitoring programs at Love Canal in Niagara Falls, New York, and in Dallas, Texas, that dealt with suspected contaminated soils. Both of these monitoring programs were conducted over a relatively short time with the collection and analysis of over 4000 soil samples. The methods employed by the Environmental Protection Agency for providing soil data that was scientifically valid and of defensible quality for each of these monitoring programs are presented. Also, methods for identifying data bias, its precision and its uncertainty are identified.

1. Introduction

In recent years there has been an increasing concern about the growing exposure of humans to potentially toxic chemicals. This potential exposure in many instances is due to initial contamination of the soil system followed by various transport routes to man. As such, demands have increased for the design of monitoring systems that will identify and define the concentrations and the chemical and physical behavior of pollutants in soils.

Environmental scientists dealing with the monitoring and sampling aspects of contaminated soils are confronted with a wide variety of problems. These problems have ranged from lack of adequately tested sampling designs and sampling procedures for collecting meaningful information for use in enforcement actions, or even for monitoring, modeling and migration studies, to a complete lack of appropriate analytical methods for obtaining adequate data. These problems, until recent years, existed because of a lack of appropriate research. For example, as Mason (1982) reports, the knowledge concerning soil variability, judged to be one of the key characteristics of most soil systems and essential in the design of monitoring strategies, had changed very little until about two decades ago. The implementation and selection of suitable soil sampling methods is another critical area in which investigative studies are required. For instance, many soils contaminated with hazardous materials contain volatile compounds. However, the sampling methods that have been employed to collect soils for the determination of volatile components, have to a large degree, been unsatisfactory. Sampling problems encountered have included the loss of volatile chemicals during sample

transfer, sample transport from field to laboratory, sample compositing and mixing, and in removing or opening sample container seals to collect the volatile components. Similar problems also exist for other soils contaminants.

The increased contribution of soil scientists in the design and implementation of soil monitoring strategies for dealing with hazardous materials evolved to a large degree with the advent of the U.S. National Contingency Plan (NCP). The primary thrust of the original NCP, issued in 1968 as an interagency agreement between the Departments of Interior; Defense; Transportation; Health, Education and Welfare; and the Office of Emergency Preparedness was directed primarily to oil spills. From the NCP's inception in 1968 until final passage in 1982 a number of important changes affecting the role of terrestrial scientists occurred. Perhaps the major change occurred when the NCP was revised to meet the statutory requirements of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), and section 311 of the Clean Water Act (CWA). These statutory CERCLA and CWA requirements not only provided the scientist with a means of discovering and investigating areas in which hazardous materials had been and are being disposed of, but also gave him the investigative authority. With these responsibilities came the requirement for scientists to design efficient monitoring programs that are not only economically feasible and address the required objectives, but produce decision-making data of the highest quality.

In 1980, at Love Canal, located in Niagara Falls, New York, and again in 1982 in Dallas, Texas, environmental scientists at the U.S. Environmental Protection Agency's (U.S. EPA) Environmental Monitoring Systems Laboratory in Las Vegas (EMSL-LV) conducted soil monitoring programs that were designed, in part, to assess and identify the geographical extent of elemental lead contamination. In both cases the data obtained were used for making decisions concerning the mitigation of identified contaminants. This paper describes the soil handling methods selected for each of the two monitoring programs; the Quality Assurance/Quality Control (QA/AC) samples and procedures utilized, and the data validation methods used.

2. Soil Handling Methods

2.1. Love Canal

In response to a presidential order declaring a state of emergency at Love Canal due to a potential public health problem (Love Canal, 1978), plus the great anxiety experienced by local residents living near this hazardous waste site, the EPA was required to provide a rapid assessment of conditions. The monitoring program designed and implemented by EPA was therefore limited to a 3-month sampling period. During this period over 1000 soil samples were collected from over 170 geographical locations that had been selected for sampling. Because the monitoring strategy employed and the criteria used for selecting these sampling locations have been previously described by Schweitzer (1982) and by U.S. EPA (1982), the methods employed and the techniques used for this monitoring program will not be presented in great detail.

To address, the soil variability problem at Love Canal and to collect as representative a sample as possible, five subsamples were collected at each location. All five subsamples were placed in a stainless steel container and mixed by hand until suitably homogenized. For the elemental analysis an aliquot consisting of a minimum of 200 g was placed in a pre-cleaned glass sample container. These samples were handled under chain-of-custody procedures until received at the analytical laboratory.

The analytical method selected for the elemental analysis was Atomic Absorption Spectroscopy (AAS) according to the procedures identified by Kopp and McKee (1978). The sample preparation steps prior to AAS analysis included drying, pulverizing, mixing, digesting with nitric acid (HNO3) and oxidizing with hydrogen peroxide (H2O2).

2.2. Dallas

The EPA's monitoring program conducted in Dallas was in response to a potential public health problem in the form of elevated blood levels in pre-school aged children living near and in the immediate vicinity of two secondary lead smelters. This program involved the collection of approximately 3000 soil samples in the vicinity of these two smelters over a 90-day period. The data obtained from these samples were used for two purposes; to determine if statistical correlation exists between blood lead levels and soil lead levels and to delineate, by the application of geostatistics, geographical areas of lead contamination in the vicinity of each smelter.

Similar to the Love Canal monitoring program the Dallas sampling strategy and sampling methods were previously described (Brown *et al.*, 1983). The four subsamples collected at each sampling location at the Dallas Lead smelter sites to minimize soil variability were mechanically mixed in a laboratory to achieve homogeneity prior to their submission to the analytical laboratory. These samples were also handled under chain-of-custody procedures.

The analytical methods (Brown *et al.*, 1983) selected for the lead analyses of the Dallas soils were similar to those used for the Love Canal soils. The primary differences between the two methods were that for the Dallas soils, 5 g instead of 2 g samples were nitric-acid digested and the Love Canal samples were further treated with hydrogen peroxide.

2.3. Sample homogeneity

To evaluate the mixing procedures used to obtain sample homogeneity for the two soil monitoring programs, aliquots were collected from the hand- and laboratory-mixed samples. Five-gram (5 g) or 2 g portions were taken from each of the aliquots and analyzed for lead. The mean value and the relative standard deviation (%) were calculated for each of the aliquots. The relative standard deviation for lead in the Dallas soils ranged from a low of 0.34% to a high of 18.0%. For the Love Canal soils, the range varied from a low near 10.0% to a high that exceeded 50.0%. This fairly high relative standard deviation for lead in the Love Canal soils not only reflects the method of mixing but also the amount of soil collected per sampling location. At Love Canal each soil

sample exceeded 30 kg, whereas the amount of soil collected at each sampling location in Dallas was approximately 150 g.

3. Quality Assurance/Quality Control Procedures

The primary objective of the Love Canal–Dallas Lead QA/QC programs was to provide monitoring data that possessed the maximum accuracy attainable. This includes precision, which addresses variability, specifically the identification of the range of values that may be encountered in identically repeated measurements, and recovery which includes both the qualitative and quantitative analytical measurements.

To achieve this objective, to handle the large volume of samples efficiently and economically and yet to provide high quality decision-making data, a large number of QA/QC samples were processed in conjunction with the field samples. In addition, a number of QA/QC sampling and analytical procedures were incorporated. The numbers and types of QA/QC samples and procedures implemented, as identified in Table I, also proved to be satisfactory for assessing the adequacy of the analytical

TABLE I

QA/QC samples and procedures

Procedure	Comment	Corrective action
1. Field Blanks: Samples of distilled deionized water (DDI) that were used to rinse sample equipment.	1. One field blank was prepared by each sampling team per day for the detection of accidental or incidental contamination due to soil sampling equipment.	1. Check preparation procedure. 2. Check sampling and analytical procedures. 3. Invalidate results.
2. Sample Bank Blanks: Field blank samples used to rinse laboratory soil handling equipment, e.g., drying dishes, soil sieve, mill jar.	1. One sample bank blank was prepared for every 40 soil samples processed.	1. Check preparation procedure. 2. Check sampling and analytical procedures. 3. Invalidate results.
3. Duplicate Sample: An additional soil sample collected at a designated sampling location.	1. One duplicate sample was collected for every 20 soil samples collected. Duplicates were used to determine total random error.	1. None.
4. Triplicate Sample: A field sample that has been laboratory or field processed and divided into three equal portions.	1. One triplicate sample was prepared for each 20 soil samples processed. Triplicate samples provided a blind duplicate for analyses and a replicate for the referee laboratory. Triplicate samples were used to determine interlaboratory precision.	1. None.

Table I (continued)

Procedure	Comment	Corrective action
5. Calibration and Standardization: Calibrate and standardize instrument as per standard methods. Analyze a Calibration Control Standard (CCS) immediately after calibration and after the analysis of every 20 soil samples.	1. Calculate CCS as percent true value. Maintain control chart for CCS and develop the 99 and 95% confidence limits (control and warning). Acceptable range is the lesser of 95 to 105% or the 99% confidence limits.	1. Analyze a second Calibration Control Sample. 2. Recalibrate and restandardize instrument (including fresh stock and working standard, if necessary). 3. Invalidate results and repeat analyses.
6. Reagent Blank: Samples of reagents used in the analytical procedures.	1. No significant lead concentrations should be found ($\leq 0.25\ \mu g\ ml^{-1}$). Prepare and process one reagent blank for every 20 soil samples.	1. Check for reagents and glassware contamination. 2. Correct for blank.
7. Spiked Extract: Prepare one spike extract for every twenty field sample extracts, add standard lead nitrate solution. Spike level should be approximately equal to the endogenous level or ten times the instrument detection limit.	1. Recovery for lead should be 90–110%.	1. Repeat on two additional sample extracts. 2. If either or both of the spikes are outside control limits, analyze 20 extracts by method of standard additions (Kopp and McKee, 1978).
8. Residue Reextraction: For every twenty soil samples extracted, reextract and analyze one sample residue to determine extraction efficiency.	1. Calculate % extraction (first extraction). The initial limit should be 90%.	1. Check analytical procedure. 2. Analyze laboratory control standard. 3. Invalidate results.
9. 'Total Recoverable Analysis': For every forty soil samples, a second aliquot of a sample is reextracted and analyzed using the 'Total Recoverable' analysis method (Brown *et al.*, 1983).	1. Compare 'Total Recoverable' results to extraction method results. Results should agree within 10%.	1. Check analytical procedure. 2. Analyze a second sample by 'Total method'. 3. Invalidate results.
10. Laboratory Control Standard (LCS): To determine method bias analyze one LCS for every twenty soil samples. If the first 10 results meet the control limits, reduce to one per shift. LCS is to consist of 5 grams of NBS 1645 River Sediment.	1. Calculate – LCS as percent true value. Maintain a control chart for LCS and develop the 99 and 95% confidence limits (control and warning). The acceptable recovery range is 90–110% or the 99% confidence limits, whichever is less.	1. Check Analytical Procedure. 2. Analyze second LCS sample. 3. Invalidate results.

Table I (continued)

Procedure	Comment	Corrective action
11. Split Extract Analysis: To determine injection and instrument reproducibility analyze a second aliquot of one extract. Process one split for every twenty soil sample extracts.	1. A precision of 3% is expected.	1. Recalibrate as in No. 5. 2. Analyze a second sample extract in duplicate.
12. Spiked Sample Analysis: For every twenty field samples, a duplicate portion of one sample is spiked with lead at a level approximately equal to the endogenous level but at least 10 × detection limit.	1. Calculate % recovery. Maintain control charts on the percent recovery and develop the 99 and 95% confidence limits (control and warning). Acceptable range for lead recovery is 90 to 110%, or the 99% confidence limit.	1. Check analytical procedure. 2. Repeat spiked sample analysis on an additional sample.

procedures as well as identifying the effectiveness of equipment decontamination procedures and, thus, identifying potential sample contamination.

4. Data Validation

Validation of sample data sets consisted of checking and assessing the accompanying QA/QC data. The criteria for the three QA/QC samples and procedures used to validate all data included:

Samples and procedures	Criteria
1. Reagent Blanks	Concentrations had to be less than 0.25 μg ml^{-1}.
2. Calibration Check Standards	Recovery must be between 95% and 105% of the known value for either the first analysis or the first re-check analysis.
3. Laboratory Control Standard	Recovery must be betwen 90% and 110% of the known value for either the first analysis or the first re-check analysis.

5. Assessing Uncertainty

Data produced by any sampling and analyzing system are affected by two types of errors; random and systematic. The accuracy of any one result then, is a function of the bias (due to systematic error) and precision (due to random error) of the collection and analysis methodology. Bias has at least two components, associated with extraction and instrument efficiency, and is assessed by the mean recovery of Calibration Check Standards and Laboratory Control Standards (LCS). The LCS checks overall bias for the system; the Calibration Check Standard determines the instrumental bias.

Total random error can be assessed by analyzing duplicate samples, but it includes errors due to sample collection, sample homogeneity, sample extraction, sample composition (matrix effects) and instrumental reproducibility. These errors can be evaluated by the use of the other QC procedures stated above and are assessed by calculating the standard deviations of the various analyses.

The accuracy of analysis, i.e., bias and precision, are evaluated separately below for the two types of samples, using the following equations:

$$\text{Recovery} = \text{Amount Found}/\text{Known Amount} \tag{1}$$

$$\text{Bias (B)} = \text{Recovery} - 1\,. \tag{2}$$

Difference (D) = $|x_1 - x_2|$ where x_1 and x_2 are the analytical results of paired analyses and the average is:

$$\overline{D} = \frac{\Sigma_i |X_1 - X_2|_i}{n} \tag{3}$$

and the precision is:

$$\overline{S}_x = \text{Precision} = 0.8862\,\overline{D} \tag{4}$$

where 0.8862 converts the range of two results to the standard deviation (Natrella, 1963).

If components errors are used to assess total random error, then

$$\overline{D} = (\overline{D}_1 + \overline{D}_2 + \ldots)/n \quad \text{and}$$

$$S_x = \text{Precision} = 0.8862\,[(\overline{D}_1^2 + \overline{D}_2^2 + \ldots) + S_1^2 + S_2^2 + \ldots]^{1/2}\,. \tag{5}$$

Equation (3) is suitable for use on results where the concentration varies over a very narrow range. If the concentrations found vary by an order of magnitude or more, then the difference should be normalized by dividing by the average of the two values and the precision is expressed as the coefficient variation (CV) which is $S/\bar{x}$

$$\overline{D}_n = (\Sigma_n |x_1 - x_2|_i) \div \frac{x_1 + x_2}{2}$$

$$= 2\Sigma_n |x_1 - x_2|_i \div (x_1 + x_2) \tag{6}$$

$$CV = 0.8862\,\overline{D}_n\,. \tag{7}$$

6. Results

6.1. Love Canal

Three analytical laboratories were involved in the analysis of soil samples collected during the Love Canal study. Each of the laboratories was sent a Performance

Evaluation (PE) sample prior to the start of soil analysis. These samples consisted of priority pollutants (inorganics) in reagent water because no suitable soil reference material was available. Each of the soil samples was analyzed for 14 inorganic elements by AAS but only the results of analysis for lead are considered here.

Since the three laboratories achieved acceptable results on the PE samples and produced valid results for the soil analyses, all of the QA/QC results are combined in the following tabulation.

QC sample	No.	Mean	S
Reagent Blank ($\mu g\, l^{-1}$)	18	0	
Field Blank ($\mu g\, l^{-1}$)	18	0	
Laboratory Control Standard	28	100%	12%
Spiked Sample (Recovery)	58	96.7%	18.3%
Duplicate Sample (CV)	16	0.235	0.21
Duplicate Aliquot (CV)	27	0.237	0.21
Triplicate Analyses (CV)	7	0.30	0.266

(1) Bias: The data from the field and reagent blanks indicate no measurable contamination occurred during sample collection and analysis: the detection limit was 0.2 $\mu g\, g^{-1}$. The percent recovery for the LCS indicates no bias existed in the analytical method. The spike sample results suggest that errors in the spiking process combined with errors in the sample aliquot, cf., duplicate aliquot standard deviation, yield a random error no larger than does duplicate sample analyses.

(2) Precision: The standard deviations of duplicate sample and duplicate aliquot analyses suggest that sampling error was large and that the samples were very inhomogeneous. This is not surprising, considering the number of soil layers represented in a two meter core. The total random error, based on duplicate analyses, is 21% for intralaboratory precision and, based on triplicate analyses, is 27% for interlaboratory precison.

(3) Uncertainty: The data for bias and precision can be combined to yield the uncertainty for any reported concentration by use of the following equation:

$$U = (1 + B \pm 2C), \tag{8}$$

where B is the bias, C is the standard deviation or coefficient of variation as appropriate, and 2 converts these to the 95% confidence limits.

Using the bias and precision estimates above, the 95% bounds on a reported value, X, are:

$$0.58X \text{ to } 1.42X \text{ (single operator), and } 0.46X \text{ to } 1.54X \text{ (multi-operator)}.$$

The laboratory performing most of the analyses performed noticeably better than this with 95% bounds being: $0.77X$ to $1.33X$. Since all three laboratories reported similar recoveries for added analytes, the difference was due to the higher precision achieved by that laboratory.

6.2. DALLAS

The limit of detection, approximately 0.25 μg ml^{-1}, was tested on about 10 blank analyses using a more sensitive absorbance wavelength for lead on the AAS. The result was less than 0.1 μg ml^{-1}, or 2 μg g^{-1}, for sample analysis. This suggests that most of the blank analyses were less than 2 μg g^{-1}, but this cannot be stated with any confidence. The results of the QC analyses were as follows:

QC sample	No.	Mean	S
Calibration Check Standard	150	101.5%	2.6%
Laboratory Control Standard	147	101.2%	4.1%
Field Blank (μg ml^{-1})	76	<0.25	
Sample Bank Blank (μg ml^{-1})	77	<0.25	
Reagent Blank (μg ml^{-1})	148	<0.25	
Re-extraction Analysis	17	1.7%	1.4%
Total Recoverable	144	99.8%	8.0%
Split Extract (CV)	147	0.0089	0.0079
Spiked Extract	147	99.4%	5.0%
Spiked Sample	147	100.4%	5.1%
Duplicate Aliquot (CV)	134	0.053	0.047
Duplicate Sample (CV)	129	0.189	0.168
Triplicate Analysis (CV)	220	0.144	0.128

(1) Bias: The percent recoveries indicated above for the Calibration Check Standards and LCS's suggest a small positive bias for the method of soil analysis, due principally to instrument reproducibility. The result, using Equation (2), is:

$$\text{Bias} = \text{Recovery} - 1 = 1.012 - 1 = 0.012 .$$

(2) Precision: The recovery of the analyte by the analytical method compared to the 'total' recoverable method was essentially equal and re-extraction of the residue left from the initial extraction indicated an additional $1.7 \pm 1.4\%$ recovery, also essentially equivalent. Furthermore, the results of the three types of blank analyses indicate no measurable contamination from reagents, sample collection, or sample preparation. The remaining random errors are evaluated below. Because of the wide range of concentration of lead in the samples, the coefficient of variation is used, Equation (7).

Precision (total random error) from Duplicate Sample analysis:

$$CV = 0.168 \text{ or } 16.8\% \text{ of sample concentration} .$$

The component random errors, summed as per Equation (5), are:

$$S_x = (0.0079^2 + 0.05^2 + 0.051^2 + 0.0472)^{1/2} = 0.085 .$$

These random errors suggest that reproducibility errors (0.0079) are small and that extract matrix, sample matrix, and sample homogeneity errors are equivalent. The sum of these errors is about half the total random error so the sampling error is essentially equal to all other errors combined.

Interlaboratory precision as calculated from the results of triplicate analyses, using Equation (7), is:

$$\text{Precision} = CV = 0.128 \text{ or } 12.8\% \text{ of sample concentration},$$

(3) Uncertainty: For soil analyses, using Equation (8) and the bias and CV derived above, the 95% confidence bounds on a reported value, X, are:

$$\text{Soil result will lie between } 0.676X \text{ and } 1.348X \ \mu g\, g^{-1}.$$

7. Conclusions

As the environmental scientist becomes a major contributor in the design and implementation of monitoring strategies, his responsibility for providing defensible data is essential. To satisfy this requirement, an appropriate QA/QC plan that addresses and identifies all aspects of the monitoring strategies sampling, analytical, data validation, and data reporting procedures is required. The QA/QC plan must ensure and document that all data collected, whether used for research or for monitoring purposes, is scientifically valid, defensible and of known precision and accuracy. The plans described herein, though designed for analysis of lead in soil, can be used as a guide for other sampling and analysis protocols.

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